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RF:ej 1/7/02 128.833USN

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Date of Mailing: 7 January 2002

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE
(DO/EO/US) CONCERNING FILING UNDER 35 U.S.C. 371

Attorney Docket No.: 128.833USN

Int'l. Application No.: PCT/SE00/01435
Int'l. Filing Date: 5 July 2000
Priority Date Claimed: 6 July 1999
Title of Invention: SYSTEM AND METHOD FOR OXYGEN
DELIGNIFICATION OF PULP MADE FOR
LIGNOCELLULOSIC MATERIAL
Applicant(s) for DO/ES/US: Hakan Dahloff and Martin Ragnar

Applicant herewith submits to the United States
Designated/Elected/Office (DO/EO/US) the following items and
other information:

1. ☒ This is a FIRST submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a SECOND or SUBSEQUENT submission of items concerning a filing under 37 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office(RO/US).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☐ have not been made and will not be made.
9. ☒ An (unsigned) oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
11. ☐ An Information Disclosure Statement under 37 C.F.R. 1.97 and 1.98.
12. ☐ An assignment document for recording. A cover sheet in

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compliance with 37 C.F.R. 3.28 and 3.31 is included.

13. ☒ A FIRST preliminary amendment.
14. ☐ Applicant qualifies for Small Entity Status (37 C.F.R. 1.9(f) and 1.27(b)).
16. ☐ Other items or information: (if any)
17. ☒ Basic National Filing Fee of \$1040.00 is submitted (Neither international preliminary examination fee (37 C.F.R. 1.482) nor international search fee 37 C.F.R. 1.44.5(a)(2) paid to U.S.P.T.O.).

CLAIMS AS FILED			
For	Number Filed	Number Extra	Basic Fee \$1040.00 Rate
Total Claims	? - 20	= 0	x \$18.00 = \$0.00
Ind. Claims	? - 3	= 0	x \$84.00 = \$0.00

19. ☐ Reduction by 1/2 for filing by small entity, if applicable. Applicant qualifies as small entity.
TOTAL FILING FEE: \$520.00.
20. ☐ Fee for recording the enclosed assignment (37 C.F.R. 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 C.F.R. 3.28, 3.31). \$40.00 per property.
21. ☒ A check in the amount of \$1040.00 to cover the above fees is enclosed.
23. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 06-0243.

Respectfully submitted,

Rolf Fasth

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APR. 15. 7:00AM

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RF: 4/15/02 128.033USN

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of

Art Unit

Hakan Dahloff and Martin
Ragnar.

Serial No. 10/030,730

Filed: January 7, 2002

For: SYSTEM AND METHOD FOR
OXYGEN DELIGNIFICATION
OF PULP MADE FOR
LIGNOCELLULOSIC MATERIAL

Examiner:

Date: 15 April 2002

SUPPLEMENTAL PRELIMINARY AMENDMENT

Assistant Commissioner for Patents
Washington, DC 20231

Preliminary to examination, please amend the above-
identified patent application as follows:

In the Specification:

Please add the following paragraph directly under the
title:

--Prior Applications

This application is a U.S. national phase application
based upon International Application No. PCT/SE00/01435, filed
5 July 2000; which claims priority from Swedish Application
No. 9902586-8, filed 6 July 1999.--

APR. 15. 2002 7:00AM

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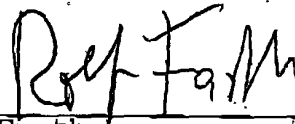
REMARKS

Reconsideration of the application is respectfully requested. The application has been amended to include all earlier-filed applications for which priority has been claimed in accordance with 37 CFR 1.78. A copy of International Application No. PCT/SE00/01435 and a certified copy of Swedish Application No. 9902586-8 are being submitted herewith.

The application is submitted to be in condition for allowance, and such action is respectfully requested.

Respectfully submitted,

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1003073 NO. 307030730

JC13 Rec'd PCT/PTO 07 JAN 2002

RE:aj 1/7/02 128.933USN

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of

Art Unit

Hakan Dahloff and Martin
Ragnar

Serial No.

Filed: Herewith

For: SYSTEM AND METHOD FOR
OXYGEN DELIGNIFICATION
OF PULP MADE FOR
LIGNOCELLULOSIC MATERIAL

Examiner:

Date: 7 January 2002

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents
Washington, DC 20231

Preliminary to examination, please amend the above-
identified patent application as follows:

In the specification:

Please delete the entire patent specification and
replace with the patent specification as shown in Appendix A.

In the claims:

Please cancel claim 1-9 and replace with the claims, as
outlined in Appendix B.

In the abstract:

Please delete the original abstract and replace with
the abstract shown in Appendix C.

REF: 1/7/02 128.633USN

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Remarks

Reconsideration of the application is respectfully requested. The original patent specification has been revised, as shown in Appendix A. The revised specification contains no new matter.

The claims have been rewritten, as shown in Appendix B. The rewritten claims contain no new matter.

The abstract has been rewritten, as shown in Appendix C. The rewritten abstract contains no new matter.

The application is now submitted to be in condition for allowance, and such action is respectfully requested.

Respectfully submitted,

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APPENDIX A

Rewritten Specification

System and process for the oxygen delignification of pulp consisting of lignocellulose-containing material.Technical Field

5 The present invention relates to a system and a process for oxygen delignification.

Background and Summary of the Invention

10 A number of different processes for oxygen delignification are known. For example, US Patent No. 4.259.150 presents a system with multistage oxygen bleaching in which, in each stage, the pulp is first mixed to a lower consistency with O₂, water and NaOH, followed by a thickening back to the consistency level which the pulp had prior to the stage in
15 question. The aim is to obtain an economic, chlorine-free bleaching with high yield. At the same time, the kappa number can be lowered, by means of repeated stages, from 70 down to 15 or even less than 15.

20 Swedish Patent C,467.582 presents an improved system for the oxygen bleaching of pulp of medium consistency. By means of controlling the temperature in an optimized manner, an oxygen bleaching takes place in a first delignification zone at a low temperature, with this being followed by a second delignification zone at a temperature which is 20-40 degrees higher. The aim is
25 to obtain an improved yield and an improved viscosity, while retaining the dwell time, in association with industrial use.

Other variants of oxygen delignification in two stages have also been patented in addition to Swedish Patent No. C,467.582. Swedish Patent No. C,505.147 presents a process in
30 which the pulp should have a high pulp concentration in the range of 25-40% in the first stage and a concentration of 8-16% in the

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second stage, at the same time as the temperature in the second stage should be higher than, or equal to, the temperature in the first stage, in line with the temperature difference which is recommended in Swedish Patent No. C,467.582. The advantages of the solution in accordance with Swedish Patent No. C,505.147 are stated to be the possibilities of admixing more oxygen in the first high-consistency stage without there being any risk of channel formation but where, at the same time, unused quantities of oxygen can be bled off after the first stage in order subsequently to be admixed in a second mixer prior to the second stage.

Swedish Patent No. C,505.141 presents a further process which is an attempt to circumvent Swedish Patent No. C,467.582, since that which it is sought to patent is stated to be that a temperature difference between the stages does not exceed 20 degrees, i.e., the lower suitable temperature difference patented in SE,C,467.582, but that a temperature difference should nevertheless be present. In addition to that, it is stated that a) the pressure should be higher in the first stage and b) that the dwell time is short in the first stage, i.e., in the order of magnitude of 10-30 minutes, and also c) the dwell time in the second stage is longer, i.e., in the order of magnitude of 45-180 minutes.

A lecture entitled "Two stage MC-oxygen delignification process and operating experience" which was given by Shinichiro Kondo from the Technical Div. Technical Dept. OJI PAPER CO. Ltd. At the 1992 Pan-Pacific Pulp & Paper Technology Conference, 99 PAN-PAC PPTC, Sept. 8-10, Sheraton Grande Tokyo Bay Hotel & Towers, presents a successful installation which was constructed with two-stage oxygen delignification in 1986 in a plant in Tomakomai.

In this OJI PAPER plant in Tomakomai, the pulp was fed, with a pressure of 10 bar, to a first oxygen mixer (+ team)

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followed by an after-treatment in a pre-retention tube (pre-reactor), with a 10 minute dwell time in which the pulp pressure is reduced to a level of about 8-6 bar due to pipe losses, etc. After that, the pulp was fed to a second oxygen mixture followed by an after-treatment in a reactor at a pressure of 5-2 bar and with a dwell time of 60 minutes. It was stated at this point that preference would have been given to having a pre-retention tube which would have given a dwell time of 20 minutes but that it was not possible to construct this due to lack of space. The OJI PAPER stated that, by using this installation, they had succeeded in obtaining an increase in kappa reduction at a lower cost in chemicals and with the pulp viscosity being improved.

Most of the prior art has consequently been directed towards a higher pressure in the first reactor at a level of about 6(8)-10 bar. A pressure in the first reactor of up to 20 bar has even been discussed in certain extreme applications. This results in it being necessary to manufacture the reactor spaces which are required for the first delignification zone such that they can cope with these high pressure levels, with a consequent requirement for substantial material thickness and/or good material qualities, which in turn result in an expensive installation.

In pulp suspensions in industrial production processes, there are large quantities of readily oxidizable constituents/structures which already react under modest process conditions. It is therefore advantageous, in a first stage, to add oxygen in quantities which are such that this part of the pulp which is relatively easily oxidized is allowed to oxidize/react first of all. Severe problems arise if an attempt is made to compensate for this by over-adding oxygen since there is the immediate danger of canalization problems, as mentioned in Swedish Patent No. C,505.147.

One object of the present invention is to avoid the

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disadvantages of the prior art and to obtain an oxygen delignification which gives increased selectivity. The present invention permits an optical practical application of the theories regarding a first rapid phase and a second slower phase during the oxygen delignification process, with the optimal reaction conditions being different between the phases.

At the high hydroxide ion concentrations and high oxygen partial pressures which are conventionally employed in the first stage, the carbohydrates are attacked more than is necessary, thereby impairing the quality of the pulp. A lower oxygen partial pressure, and preferably a lower temperature as well, in the first stage as compared with the second stage decreases the rate of reaction for the breakdown of carbohydrates more than it decreases the rate of reaction for the delignification, leading in turn to an increase in the total selectivity on the pulp after the two stages.

Another object of the present invention is to allow a simpler and cheaper process installation in which at least one pressure vessel, in a first delignification zone, can be manufactured using thinner material and/or using a lower material quality which is suitable for a lower pressure class.

Yet another object is also to make it possible to use steam at moderate pressure especially when there is a need to increase the temperature substantially between the first and second stage and when the pressure in the second stage is considerably higher than that in the first stage. In most cases, the supply of medium-pressure steam and low-pressure steam is very good in a pulp mill whereas high-pressure steam is in short supply due to the large number of processes which require high-pressure steam. This also makes it possible to convert existing single-vessel delignification systems where, with the previously the prior art for converting to a two-stage design, a restriction has been imposed by the fact that the prevailing pressure in the

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plant's steam grid has not enabled a sufficiently large quantity of steam to be admixed with the pulp in order to achieve the desired temperature in the second delignification stage.

Yet another object is to optimize the mixing process in each position such that only that quantity of chemicals/oxygen is added which is consumed in the subsequent delignification zone and where the admixture of chemicals/oxygen does not need to compete with the simultaneous admixture of steam for the purpose of increasing the temperature to the desired level. In this way, it is possible to dispense with bleeding systems for overshooting quantities of oxygen at the same time as it is possible to reduce the total consumption of oxygen, which in turn reduces the operating costs for the operator of the fibre line and thus shortens the pay-off time. At the same time, it is possible to select a smaller size of dynamic mixer for admixing chemicals, which mixer is dimensioned solely for the volumes of chemicals which are actually being admixed.

Yet another object is to increase, in an oxygen delignification system having a certain total volume of the first and second stages, a so-called H factor by operating the first stage for a short time at low temperature and operating the second stage for a longer time at a higher temperature. Thus, in connection, for example, with conversions of existing single-vessel oxygen delignification stages, a simple conversion, including a small pre-reactor and a modest increase in the reaction temperature in the existing reactor, can increase the H factor and at the same time improve the selectivity over the oxygen stages.

30 Brief Description of the Drawings

Fig. 1 shows a system for oxygen delignification in two stages in accordance with the invention; and

Fig. 2 diagrammatically shows the kinetics of the

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oxygen delignification and the advantages which are gained relative to the prior art with regard to reduction in kappa number and an increased H factor.

5 Detailed Description

Fig. 1 shows an installation, according to the present invention, of a system in an existing plant in which the oxygen delignification process needed upgrading.

10 An existing first MC pump 1 (MC = medium consistency, typically a pulp consistency of 8-18%) is connected to a tipping chute 2 for forwarding to an existing first MC mixer 3. The first mixer 3 is a so-called dynamic mixer, in which a motor-driven rotor agitates the pulp in at least one narrow fluidization gap. The dynamic mixer is preferably a mixer type
15 which corresponds to that which is shown in US433920, in which a first cylindrical fluidization zone is formed between the rotor and the housing and a second fluidization zone is formed between a radially directed rotor part and housing, which mixer is hereby introduced as a reference. A mechanical agitation is required in
20 order to obtain a uniform admixture of the chemical charge in question in the whole of the pulp suspension, with the aim of the pulp being bleached/treated uniformly throughout the whole of the volume of the pulp.

An admixture of chemicals, chiefly oxygen, takes place
25 in the first MC mixer 3, after which the pulp was, in the existing system, fed to an oxygen reactor 6. The combination of a first MC pump 1 followed closely by an MC mixer 3 can be termed a perfect pair. This is the case since the pump primarily pressurizes the pulp flow to a given degree, thereby facilitating
30 a finely divided supply of the oxygen to the MC mixer which follows directly thereafter.

In accordance with the invention, an upgrading of the oxygen delignification process is achieved by introducing a

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static mixer 8, i.e., a non-rotating or mechanically agitating mixer 8 for increasing the temperature by means of adding steam. The static mixer 8 is preferably of a construction which has been shown in SE,C,512.192 (= PCT/SE00/00137), where steam is conducted in as thin jets through a number of holes which are uniformly distributed over the periphery of a pulp-conveying pipe, which mixer is hereby introduced as a reference.

The static mixer 8 is arranged directly after the oxygen reactor 6 and followed by a second MC pump 4 and a second agitating MC mixer 5, of the same type as the mixer 3, which acts directly after the MC pump 4. The system is assembled such that the coupling pipe 6 forms a first delignification zone between the outlet of the first MC mixer 3 and the inlet of the non-rotating mixer 8, which zone gives rise to a dwell time R_T of 2-20 minutes, preferably 2-10 minutes and even more advantageously 3-6 minutes.

The second MC pump 4 is controlled such that the resulting pressure in the dwell line 6 is preferably in the interval 0-6 bar, preferably 0-4 bar. Preferably, the second pump 4 is controlled by means of its rotational speed being controlled by a control system PC depending on the pressure which prevails, and is detected, in the first delignification zone 6.

The temperature in the whole of the first delignification zone 6 can be kept low, preferably at the level which the system allows without adding steam, but preferably with the pulp entering the first delignification zone being at a temperature of about 85°C , $\pm 10^{\circ}\text{C}$.

The non-rotating mixer 8 is connected in after the first delignification zone, as are then the second MC pump 4 followed by the second MC mixer 5. This second perfect pair combination is controlled such that the resulting pressure in the oxygen reactor 10, which forms a second delignification zone,

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reaches a level of at least 3 bars over-pressure at the top of the reactor. In conventional applications, the pressure in the second MC mixer should be at least 4 bar higher than the pressure in the first MC mixer; alternatively, the increase in pressure in the second pump should reach 4 bar. In connection with practical implementation in conventional oxygen stages, an initial pressure is obtained within the interval 8-10 bar, corresponding to the pressure at the inlet to the reactor.

In accordance with the present invention, the temperature of the pulp in the second delignification zone is increased by supplying steam to the non-rotating mixer directly after the first delignification zone and before the pressure-raising pump 4 comes into play. The steam supply is expediently controlled using a control system TC, which comprises a control valve V on the line 7 for the steam supply and a feeding-back measurement of the temperature of the pulp which is leaving the mixer. The temperature is expediently raised to a level of $100^{\circ}\text{C} \pm 10^{\circ}\text{C}$, but preferably at least 5°C higher than the temperature in the first delignification zone. As a result of the steam being added before the pulp is given the higher pressure which is required for the final phase of the delignification:

- a higher temperature can be obtained;
- the pressure of the available steam does not need to be so high; and
- the mixers for adding chemicals/admixing oxygen do not need to be burdened with a supply of steam as well, which will otherwise reduce their efficiency.

The volume of the second delignification zone, i.e., the second reactor, is expediently designed such that it is at least 10 times greater than the volume of the first delignification zone, i.e., a retention time of at least 20-200

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minutes, preferably 20-100 minutes and even more advantageously within the range 50-90 minutes.

Fig. 2 diagrammatically shows the kinetics of the oxygen delignification and the advantages with regard to the principles of kappa number reduction which are obtained relative to the prior art. Curve P1 shows the principle of a reaction course during the initial phase of the delignification. This part of the delignification proceeds relatively rapidly and is typically essentially complete after a good 20 minutes.

However, after a relatively short time, typically only 5-10 minutes, the final phase P2 of the delignification takes over and begins to dominate as far as the resulting delignification of the pulp is concerned. A typical subdivision of the delignification into two stages in accordance with the prior art is shown at line A, with stage 1 being to the left of the line A and stage 2 being to the right of the line A. It follows from this that two different dominating processes, i.e., the initial phase of the delignification on the one hand, but also its final phase, actually take place in stage 1. It can be concluded from this that it becomes impossible to optimize the process conditions in stage 1 for both these delignification phases.

Instead, a subdivision of the delignification into two stages in accordance with the invention is shown as a line B, a stage 1 is to the left of the line B and stage 2 is to the right of the line B. This makes it possible to optimize each stage for the process which dominates in the stage. The curve H_A shows the temperature integral plotted against time (the H factor) which is typically obtained when implementing a delignification process in two stages in accordance with the prior art, corresponding to the line A.

As can be seen from the figure, it is possible to use the stage subdivision in accordance with the invention to obtain

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an H factor which is higher than that which is typically obtained in current installations. This can be done without foregoing demands for high selectivity over the oxygen delignification system. The invention also opens up ways of upgrading, with a small investment, an existing 1-stage process of comparatively low selectivity to a 2-stage system of better selectivity without having to build a new large reactor or even two such reactors. According to the present invention, the initial phase of the oxygen delignification is dealt with in the pre-reactor, after which the temperature in the existing reactor can even be increased, if so required, in association with the conversion, and an increased H factor can in this way be combined with increased selectivity.

The invention can be modified in a number of ways within the context of the inventive concept. For example, the first delignification zone can consist of a pre-retention tube which is vertical but in which the pressure in some part of this pre-retention tube, including its bottom, is at least 4 bar lower than the pressure in the initial part of the second delignification zone.

Further delignification zones, or intermediate washing/bleaching or extraction of the pulp, can be introduced between the first and second delignification zones according to the invention. For example, a third perfect pair combination, i.e., a pump with a mixer following it, can be arranged between the zones. What is essential is that the first delignification zone is characterized by a lower pressure, a short dwell time and a moderate temperature, and that the concluding, final delignification zone is characterized by a higher pressure (a pressure which is at least 4 bar higher than that of the first zone), a longer dwell time (a dwell time which is at least 10 times longer than that in the first zone) and an increased temperature (a temperature which is preferably at least 5 degrees

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higher than that in the first zone).

Where appropriate, it should be possible to charge a first mixer, or an intermediate mixer in a third perfect pair combination, with oxygen, at least some part of which is blown off from the reactor 10. The economic basis for such a recovery of oxygen is poor since the cost of oxygen is relatively low.

In order to ensure optimal process conditions, one or other, preferably the second, or both of the MC pumps can be rotation speed-controlled in dependence on the pressure in the first delignification zone.

The present invention can also be modified by a number of varying additions of other chemicals either together with the oxygen or separately from the addition of oxygen, in a separate adding position, which chemicals are selected and suitable for the specific fibre line and the pulp quality in question, such as

- alkali/NaOH for adjusting the pH level to that which is suitable for the pulp quality in question,
- agents for protecting cellulose, for example $MgSO_4$ or other alkaline earth metal ions or compounds thereof;
- additions of complex agents which are performed prior to adding oxygen, with subsequent removal of precipitated metals, where appropriate,
- chlorine dioxide;
- hydrogen peroxide or organic or inorganic peracids or salts thereof;
- free-radical capturing agents, such as alcohols, ketones, aldehydes or organic acids; and
- carbon dioxide or other additives.

Where appropriate, it should also be possible to degas exhaust gases, such as residual gases, in immediate conjunction with the second pump, preferably by means of the pump being

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provided with internal degassing, preferably a pump termed a degassing pump.

While the present invention has been described in accordance with preferred compositions and embodiments, it is to be understood that certain substitutions and alterations may be made thereto without departing from the spirit and scope of the following claims.

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APPENDIX B

Substitute Claims

10. A system for oxygen delignification of pulp having a lignocellulose-containing material having a mean concentration of 8-18% pulp consistency, the oxygen delignification taking place in at least two stages and where the system comprises:

a first pump arranged to pump the pulp to a first mixer for admixing, in the first mixer, chemicals that are required for an oxygen delignification process, the first mixer being arranged in close conjunction with the first pump;

a first delignification zone arranged to receive pulp from the first mixer;

a second pump subsequent to the first delignification zone and ahead of a second delignification zone;

a third mixer arranged in close conjunction with the second pump, for admixing, in the third mixer, chemicals that are required for the oxygen delignification process;

a second delignification zone arranged to receive pulp from the third mixer;

a second mixer arranged to receive pulp from the first delignification zone, the second mixer having means for admixing steam with the pulp; and

the second pump being arranged to receive pulp after the second mixer and having a pumping effect to obtain a lower oxygen partial pressure in the first delignification zone compared with a pressure in the second delignification zone.

11. The system for oxygen delignification according to claim 10 wherein the first and third mixers are mixers using mechanical agitation and with the pulp at least partially being fluidized in gaps defined in the mixers, and the second mixer is a static mixer without mechanical agitation.

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12. The system for oxygen delignification according to claim 11 wherein the system has means for adding oxygen to the first mixer and the third mixer, respectively, and means for adding steam to the second mixer.

5

13. The system for oxygen delignification according to claim 12 wherein the second mixer has means for supplying steam in a controllable manner that is feedback-controlled depending upon a temperature of the pulp after the second mixer.

10

14. The system for oxygen delignification according to claim 13 wherein the second mixer has a pulp-conveying pipe having a number of inlet holes defined therein for receiving steam.

15

15. The system for oxygen delignification according to claim 14 wherein the steam consists of a medium-pressure steam at a pressure of 8-14 bar.

20

16. The system for oxygen delignification according to claim 12 wherein the system comprises a control system for controlling a rotational speed of the second pump depending upon a pressure in the first delignification zone.

25

17. The system for oxygen delignification according to claim 10 wherein the first delignification zone has a volume that results in a dwell time of 2-20 minutes for the pulp in the first delignification zone, the pressure in the first delignification zone is at a pressure of 0-6 bar, the second pump has a pumping effect such that a pressure in the second delignification zone reaches a level of at least 3 bars over-pressure at a top of the second delignification zone, the second delignification zone has a volume that is at least 10 times greater than the volume of the

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first delignification zone and the volume of the second delignification zone results in a dwell time of at least 20-200 minutes.

5 18. The system for oxygen delignification according to claim 17 wherein the dwell time of the first delignification zone is 2-10 minutes.

10 19. The system for oxygen delignification according to claim 17 wherein the dwell time of the first delignification zone is 3-6 minutes.

15 20. The system for oxygen delignification according to claim 17 wherein the pressure in the first delignification zone is 0-4 bar.

20 21. The system for oxygen delignification according to claim 17 wherein the dwell time of the second delignification zone is 20-100 minutes.

22. The system for oxygen delignification according to claim 17 wherein the dwell time of the second delignification zone is 50-90 minutes.

25 23. A process for oxygen delignification of pulp having a lignocellulose-containing material having a mean concentration of 8-18% pulp consistency, the oxygen delignification taking place in at least two stages, comprising:

- 30 (a) pressurizing a median concentration pulp;
(b) adding chemicals to the oxygen delignification process in a first stage;
(c) treating the pulp for 2-20 minutes under a moderate over-pressure of between 0-6 bar and at a

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first temperature in a range of 75-95°C;

- (d) mixing the pulp with steam to heat the pulp;
- (e) pressurizing the heated pulp;
- (f) adding chemicals to the oxygen delignification process in a concluding stage; and
- (g) treating the pulp for 2-200 minutes at an initial pressure of 8-10 bar corresponding to a pressure at an inlet defined in a reactor and at a second temperature in a range of 90-110°C, the treating step (c) being longer than the treating step (g) and the second temperature being higher than the first temperature.

24. The process according to claim 23 wherein step (b) comprises adding chiefly oxygen.

25. The process according to claim 23 wherein step (c) comprises treating the pulp for 2-10 minutes.

26. The process according to claim 23 wherein step (c) comprises treating the pulp for 3-6 minutes.

27. The process according to claim 23 wherein step (c) comprises treating the pulp at an over-pressure of 0-4 bar.

28. The process according to claim 23 wherein step (f) comprises adding chiefly oxygen.

29. The process according to claim 23 wherein step (g) comprises treating the pulp for 20-100 minutes.

30. The process according to claim 23 wherein step (g)

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comprises treating the pulp for 50-90 minutes.

31. The process according to claim 23 wherein the
second temperature is at least 5°C higher than the first
5 temperature.

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APPENDIX C

ABSTRACT

5 The system is for the oxygen delignification,
in at least two reaction stages, of pulp that consists of
lignocellulose-containing material having a mean concentration of
8-18% pulp consistency. The system has a first pump followed by
a first oxygen mixer that is followed by a first delignification
10 zone. The first delignification zone is followed by a second
steam mixer that is followed by a second pump that is followed by
a third oxygen mixer and a second delignification zone.

SYSTEM AND PROCESS FOR THE OXYGEN DELIGNIFICATION OF PULP CONSISTING OF LIGNOCELLULOSE-CONTAINING MATERIAL

The present invention relates to a system and a
5 process for oxygen delignification in accordance with
the preambles to patent claim 1 and patent claim 9,
respectively.

State of the art

10

A number of different processes for oxygen
delignification are known.

US,A,4.259.150 presents a system with multistage oxygen
bleaching in which, in each stage, the pulp is first
15 mixed to a lower consistency with O₂, water and NaOH,
followed by a thickening back to the consistency level
which the pulp had prior to the stage in question. The
aim is to obtain an economic, chlorine-free bleaching
with high yield. At the same time, the kappa number can
20 be lowered, by means of repeated stages, from 70 down
to 15 or even less than 15.

SE,C,467.582 presents an improved system for
the oxygen bleaching of pulp of medium consistency. By
means of controlling the temperature in an optimized
25 manner, an oxygen bleaching takes place in a first
delignification zone at a low temperature, with this
being followed by a second delignification zone at a
temperature which is 20-40 degrees higher. The aim is
to obtain an improved yield and an improved viscosity,
30 while retaining the dwell time, in association with
industrial use.

Other variants of oxygen delignification in two
stages have also been patented in addition to
SE,C,467.582. SE,C,505.147 presents a process in which
35 the pulp should have a high pulp concentration in the
range of 25-40% in the first stage and a concentration
of 8-16% in the second stage, at the same time as the

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temperature in the second stage should be higher than, or equal to, the temperature in the first stage, in line with the temperature difference which is recommended in SE,C,467.582. The advantages of the solution in accordance with SE,C,505.147 are stated to be the possibilities of admixing more oxygen in the first high-consistency stage without there being any risk of channel formation but where, at the same time, unused quantities of oxygen can be bled off after the first stage in order subsequently to be admixed in a second mixer prior to the second stage.

SE,C,505.141 presents a further process which is an attempt to circumvent SE,C,467.582, since that which it is sought to patent is stated to be that a temperature difference between the stages does not exceed 20 degrees, i.e. the lower suitable temperature difference patented in SE,C,467.582, but that a temperature difference should nevertheless be present. In addition to that, it is stated that a) the pressure should be higher in the first stage and b) that the dwell time is short in the first stage, i.e. in the order of magnitude of 10-30 minutes, and also c) the dwell time in the second stage is longer, i.e. in the order of magnitude of 45-180 minutes.

A lecture entitled "Two stage MC-oxygen delignification process and operating experience", which was given by Shinichiro Kondo from the Technical Div. Technical Dept. OJI PAPER CO. Ltd. At the 1992 Pan-Pacific Pulp & Paper Technology Conference ('99 PAN-PAC PPTC), Sept. 8-10, Sheraton Grande Tokyo Bay Hotel & Towers, presents a successful installation which was constructed with two-stage oxygen delignification in 1986 in a plant in Tomakomai.

In this OJI PAPER plant in Tomakomai, the pulp was fed, with a pressure of 10 bar, to a first oxygen mixer (+ team) followed by an after-treatment in a "preretention tube" (prereactor), with a 10 minute

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dwelt time in which the pulp pressure is reduced to a level of about 8-6 bar due to pipe losses, etc. After that, the pulp was fed to a second oxygen mixture followed by an after-treatment in a reactor at a pressure of 5-2 bar and with a dwell time of 60 minutes. It was stated at this point that preference would have been given to having a "preretention tube" which would have given a dwell time of 20 minutes but that it was not possible to construct this due to lack of space. OJI PAPER stated that, by using this installation, they had succeeded in obtaining an increase in kappa reduction at a lower cost in chemicals and with the pulp viscosity being improved.

Most of the the prior art has consequently been directed towards a higher pressure in the first reactor at a level of about 6(8)-10 bar. A pressure in the first reactor of up to 20 bar has even been discussed in certain extreme applications. This results in it being necessary to manufacture the reactor spaces which are required for the first delignification zone such that they can cope with these high pressure levels, with a consequent requirement for substantial material thickness and/or good material qualities, which in turn result in an expensive installation.

In pulp suspensions in industrial production processes, there are large quantities of readily oxidizable constituents/structures which already react under modest process conditions. It is therefore advantageous, in a first stage, to add oxygen in quantities which are such that this part of the pulp which is relatively easily oxidized is allowed to oxidize/react first of all. Severe problems arise if an attempt is made to compensate for this by overadding oxygen since there is the immediate danger of canalization problems (as mentioned in the said SE,C,505.147).

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Object of the invention

One object of the invention is to avoid the disadvantages of the prior art and to obtain an oxygen
5 delignification which gives increased selectivity. The invention permits an optical practical application of the theories regarding a first rapid phase and a second slower phase during the oxygen delignification process, with the optimal reaction conditions being different
10 between the phases.

At the high hydroxide ion concentrations and high oxygen partial pressures which are conventionally employed in the first stage, the carbohydrates are attacked more than is necessary, thereby impairing the
15 quality of the pulp. A lower oxygen partial pressure, and preferably a lower temperature as well, in the first stage as compared with the second stage decreases the rate of reaction for the breakdown of carbohydrates more than it decreases the rate of reaction for the
20 delignification, leading in turn to an increase in the total selectivity on the pulp after the two stages.

Another object is to allow a simpler and cheaper process installation in which at least one pressure vessel, in a first delignification zone, can
25 be manufactured using thinner material and/or using a lower material quality which is suitable for a lower pressure class.

Yet another object is also to make it possible to use steam at moderate pressure especially when there
30 is a need to increase the temperature substantially between the first and second stage and when the pressure in the second stage is considerably higher than that in the first stage. In most cases, the supply of medium-pressure steam and low-pressure steam is very
35 good in a pulp mill whereas high-pressure steam is in short supply due to the large number of processes which require high-pressure steam. This also makes it

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The invention is described in more detail with reference to the figures in accordance with the following figure list.

5 Figure list

Figure 1 shows a system for oxygen delignification in two stages in accordance with the invention; AND
Figure 2 diagrammatically shows the kinetics of the oxygen delignification and the advantages which are
10 gained relative to the prior art with regard to reduction in kappa number and an increased H factor.

Description of embodiment examples

15 Figure 1 shows an installation, according to the invention, of a system in an existing plant in which the oxygen delignification process needed upgrading.

An existing first MC pump 1 (MC = medium
20 consistency, typically a pulp consistency of 8-18%) is connected to a tipping chute 2 for forwarding to an existing first MC mixer 3. The first mixer 3 is a so-called dynamic mixer, in which a motor-driven rotor agitates the pulp in at least one narrow fluidization
25 gap. The dynamic mixer is preferably a mixer type which corresponds to that which is shown in US433920, in which a first cylindrical fluidization zone is formed between the rotor and the housing and a second fluidization zone is formed between a radially directed
30 rotor part and housing, which mixer is hereby introduced as a reference. A mechanical agitation is required in order to obtain a uniform admixture of the chemical charge in question in the whole of the pulp suspension, with the aim of the pulp being
35 bleached/treated uniformly throughout the whole of the volume of the pulp.

The second MC pump 4 is controlled such that the resulting pressure in the dwell line 6 is preferably in the interval 0-6 bar, preferably 0-4 bar. Preferably, the second pump 4 is controlled by means of its rotational speed being controlled by a control system PC depending on the pressure which prevails, and is detected, in the first delignification zone 6.

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The temperature in the whole of the first delignification zone 6 can be kept low, preferably at the level which the system allows without adding steam, but preferably with the pulp entering the first
5 delignification zone being at a temperature of about 85°C , $\pm 10^{\circ}\text{C}$.

The non-rotating mixer 8 is connected in after the first delignification zone, as are then the second MC pump 4 followed by the second MC mixer 5. This
10 second "perfect pair" combination is controlled such that the resulting pressure in the oxygen reactor 10, which forms a second delignification zone, reaches a level of at least 3 bars overpressure at the top of the reactor. In conventional applications, the pressure in
15 the second MC mixer should be at least 4 bar higher than the pressure in the first MC mixer; alternatively, the increase in pressure in the second pump should reach 4 bar. In connection with practical implementation in conventional oxygen stages, an
20 initial pressure is obtained within the interval 8-10 bar, corresponding to the pressure at the inlet to the reactor.

In accordance with the invention, the temperature of the pulp in the second delignification
25 zone is increased by supplying steam to the non-rotating mixer directly after the first delignification zone and before the pressure-raising pump 4 comes into play. The steam supply is expediently controlled using a control system TC, which comprises a control valve V
30 on the line 7 for the steam supply and a feeding-back measurement of the temperature of the pulp which is leaving the mixer. The temperature is expediently raised to a level of $100^{\circ}\text{C} \pm 10^{\circ}\text{C}$, but preferably at least 5°C higher than the temperature in the first
35 delignification zone. As a result of the steam being added before the pulp is given the higher pressure

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which is required for the final phase of the delignification:

- a higher temperature can be obtained
- the pressure of the available steam does not need to
5 be so high
- the mixers for adding chemicals/admixing oxygen do not need to be burdened with a supply of steam as well, which will otherwise reduce their efficiency.

The volume of the second delignification zone,
10 i.e. the second reactor, is expediently designed such that it is at least 10 times greater than the volume of the first delignification zone, i.e. a retention time of at least 20-200 minutes, preferably 20-100 minutes and even more advantageously within the range 50-90
15 minutes.

Figure 2 diagrammatically shows the kinetics of the oxygen delignification and the advantages with regard to the principles of kappa number reduction which are obtained relative to the prior art. Curve P1
20 shows the principle of a reaction course during the initial phase of the delignification. This part of the delignification proceeds relatively rapidly and is typically essentially complete after a good 20 minutes. However, after a relatively short time, typically only
25 5-10 minutes, the final phase P2 of the delignification takes over and begins to dominate as far as the resulting delignification of the pulp is concerned. A typical subdivision of the delignification into two stages in accordance with the prior art is shown at
30 line A, with stage 1 being to the left of the line A and stage 2 being to the right of the line A. It follows from this that two different dominating processes, i.e. the initial phase of the delignification on the one hand, but also its final
35 phase, actually take place in stage 1. It can be concluded from this that it becomes impossible to

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optimize the process conditions in stage 1 for both these delignification phases.

Instead, a subdivision of the delignification into two stages in accordance with the invention is shown as a line B, a stage 1 is to the left of the line B and stage 2 is to the right of the line B. This makes it possible to optimize each stage for the process which dominates in the stage. The curve H_A shows the temperature integral plotted against time (the H factor) which is typically obtained when implementing a delignification process in two stages in accordance with the prior art, corresponding to the line A.

As can be seen from the figure, it is possible to use the stage subdivision in accordance with the invention to obtain an H factor which is higher than that which is typically obtained in current installations. This can be done without foregoing demands for high selectivity over the oxygen delignification system.

The invention also opens up ways of upgrading, with a small investment, an existing 1-stage process of comparatively low selectivity to a 2-stage system of better selectivity without having to build a new large reactor or even two such reactors. According to the invention, the initial phase of the oxygen delignification is dealt with in the prereactor, after which the temperature in the existing reactor can even be increased, if so required, in association with the conversion, and an increased H factor can in this way be combined with increased selectivity.

The invention can be modified in a number of ways within the context of the inventive concept. For example, the first delignification zone can consist of a "preretention tube" which is vertical but in which the pressure in some part of this "preretention tube", including its bottom, is at least 4 bar lower than the pressure in the initial part of the second delignification zone.

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Further delignification zones, or intermediate washing/bleaching or extraction of the pulp, can be introduced between the first and second delignification zones according to the invention. For example, a third
5 "perfect pair" combination, i.e. a pump with a mixer following it, can be arranged between the zones. What is essential is that the first delignification zone is characterized by a lower pressure, a short dwell time and a moderate temperature, and that the concluding,
10 final delignification zone is characterized by a higher pressure (a pressure which is at least 4 bar higher than that of the first zone), a longer dwell time (a dwell time which is at least 10 times longer than that in the first zone) and an increased temperature (a
15 temperature which is preferably at least 5 degrees higher than that in the first zone).

Where appropriate, it should be possible to charge a first mixer, or an intermediate mixer in a third "perfect pair" combination, with oxygen, at least some
20 part of which is blown off from the reactor 10. The economic basis for such a recovery of oxygen is poor since the cost of oxygen is relatively low.

In order to ensure optimal process conditions, one or other, preferably the second, or both of the MC
25 pumps can be rotation speed-controlled in dependence on the pressure in the first delignification zone.

The invention can also be modified by a number of varying additions of other chemicals either together with the oxygen or separately from the addition of
30 oxygen, in a separate adding position, which chemicals are selected and suitable for the specific fibre line and the pulp quality in question, such as
- alkali/NaOH for adjusting the pH level to that which is suitable for the pulp quality in question,
35 - agents for protecting cellulose, for example $MgSO_4$ or other alkaline earth metal ions or compounds thereof;

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- additions of complexing agents which are performed prior to adding oxygen, with subsequent removal of precipitated metals, where appropriate,
 - chlorine dioxide;
 - 5 - hydrogen peroxide or organic or inorganic peracids or salts thereof;
 - free-radical capturing agents, such as alcohols, ketones, aldehydes or organic acids; and
 - carbon dioxide or other additives.
- 10 Where appropriate, it should also be possible to degas exhaust gases (residual gases) in immediate conjunction with the second pump, preferably by means of the pump being provided with internal degassing, preferably a pump termed a "degassing pump".

1. System for the oxygen delignification of pulp which consists of lignocellulose-containing material and whose mean concentration is 8-18%, which oxygen delignification takes place in at least two stages and where the system comprises
- a first pump (1) which is arranged to pump the pulp to a first mixer (3) for admixing in this first mixer (3) chemicals which are required for the oxygen delignification, which first mixer is arranged in close conjunction with the first pump,
 - a first delignification zone (6) which is arranged to receive pulp from the first mixer (3),
 - a second pump (4) subsequent to the first delignification zone and ahead of a second delignification zone,
 - a third mixer (5) arranged in close conjunction with the second pump, for admixing in this third mixer (5) chemicals which are required for the oxygen delignification,
 - a second delignification zone (10) which is arranged to receive pulp from the third mixer (5)
- c h a r a c t e r i z e d i n t h a t
- a second mixer (8) is arranged to receive pulp from the first delignification zone, which second mixer comprises means for admixing steam (MP steam) with the pulp,
 - the second pump (4) is arranged to receive pulp after the second mixer, and has a pumping effect such that a lower oxygen partial pressure is obtained in the first delignification zone as compared with the second delignification zone.
2. System for oxygen delignification according to Claim 1,

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characterized in that the first and third mixers (3 and 5, respectively) are mixers using mechanical agitation, with the pulp at least partially being fluidized in gaps in the mixer, and in that the
5 second mixer (8) is a static mixer without mechanical agitation.

3. System for oxygen delignification according to Claim 2,
10 characterized in that it comprises means (O₂) for adding oxygen to the first (3) and third (5) mixers, respectively, and means (MP steam) for adding steam to the second mixer (8).

15 4. System for oxygen delignification according to Claim 3,
characterized in that the second mixer comprises means for supplying steam in a controllable manner (7,V,TC), preferably feedback-controlled in
20 dependence on the temperature of the pulp after the said mixer.

5. System for oxygen delignification according to Claim 4,
25 characterized in that the second mixer (8) consists of a pulp-conveying pipe having a number of inlet holes for the steam in the wall of the pipe.

6. System for oxygen delignification according to
30 Claim 5,
characterized in that the steam consists of medium-pressure steam at 8-14 bar.

7. System for oxygen delignification according to
35 Claim 3,
characterized in that the system comprises a control system (PC) for controlling the

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rotational speed of the second pump (4) depending on the pressure in the first delignification zone (6).

8. System for oxygen delignification according to Claim 1,

characterized

- in that the first delignification zone (6) has a volume which results in a dwell time of 2-20 minutes, preferably 2-10 minutes, and even more advantageously 3-6 minutes, for the pulp in the first delignification zone,
- in that the system is adjusted such that the pressure in the first delignification zone amounts to 0-6 bar, preferably 0-4 bar,
- in that the second pump (4) has a pumping effect such that the pressure in the second delignification zone reaches a level of at least 3 bars overpressure at the top of the second delignification zone, and
- in that the second delignification zone (10) has a volume which is at least 10 times greater than the volume of the first delignification zone, i.e. has a volume which results in a dwell time of at least 20-200 minutes, preferably 20-100 minutes, and even more advantageously within the range 50-90 minutes.

9. Process for the oxygen delignification of pulp which consists of lignocellulose-containing material and whose mean concentration is 8-18%, in at least two stages,

characterized

- a) in that pulp at median concentration is pressurized, and
- b) after that, chemicals, chiefly oxygen, are added for the oxygen delignification, such that oxygen delignification takes place in a first stage in which the pulp is treated for a relatively short time, corresponding to 2-20 minutes, preferably 2-

- 10 minutes, and even more advantageously 3-6 minutes, under moderate overpressure within the interval 0-6 bar, preferably 0-4 bar, and at a moderate temperature in the range $85^{\circ}\text{C} \pm 10^{\circ}\text{C}$,
- 5 c) and in that the pulp, after the first stage, and with the pulp being at a median concentration, is first mixed together with steam for the purpose of increasing the temperature,
- d) after which there follows a first pressurization of the heated pulp,
- 10 e) and, after that, a second addition of chemicals, chiefly oxygen, for the oxygen delignification,
- f) in order, in a concluding stage, to be treated for a longer time than in the first stage, i.e. for a
- 15 time which is of the order of magnitude of 10 times longer than in the first stage, in the interval 2-200 minutes, preferably 20-100 minutes, and even more advantageously in the interval 50-90 minutes, with this stage taking place at an
- 20 initial pressure within the interval 8-10 bar, corresponding to the pressure at the inlet of the reactor, but also at a higher temperature, preferably in the range $100^{\circ}\text{C} \pm 10^{\circ}\text{C}$, but preferably at least 5°C higher than the
- 25 temperature in the first stage.

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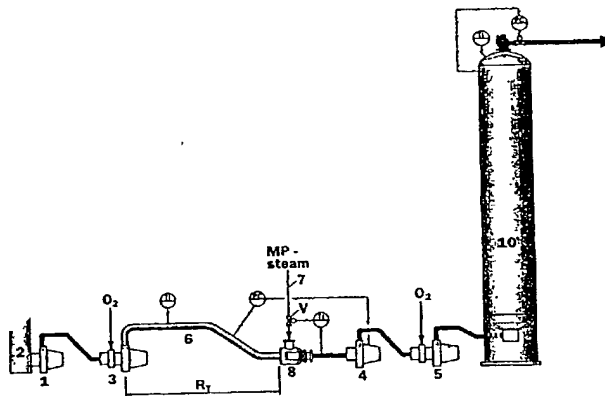
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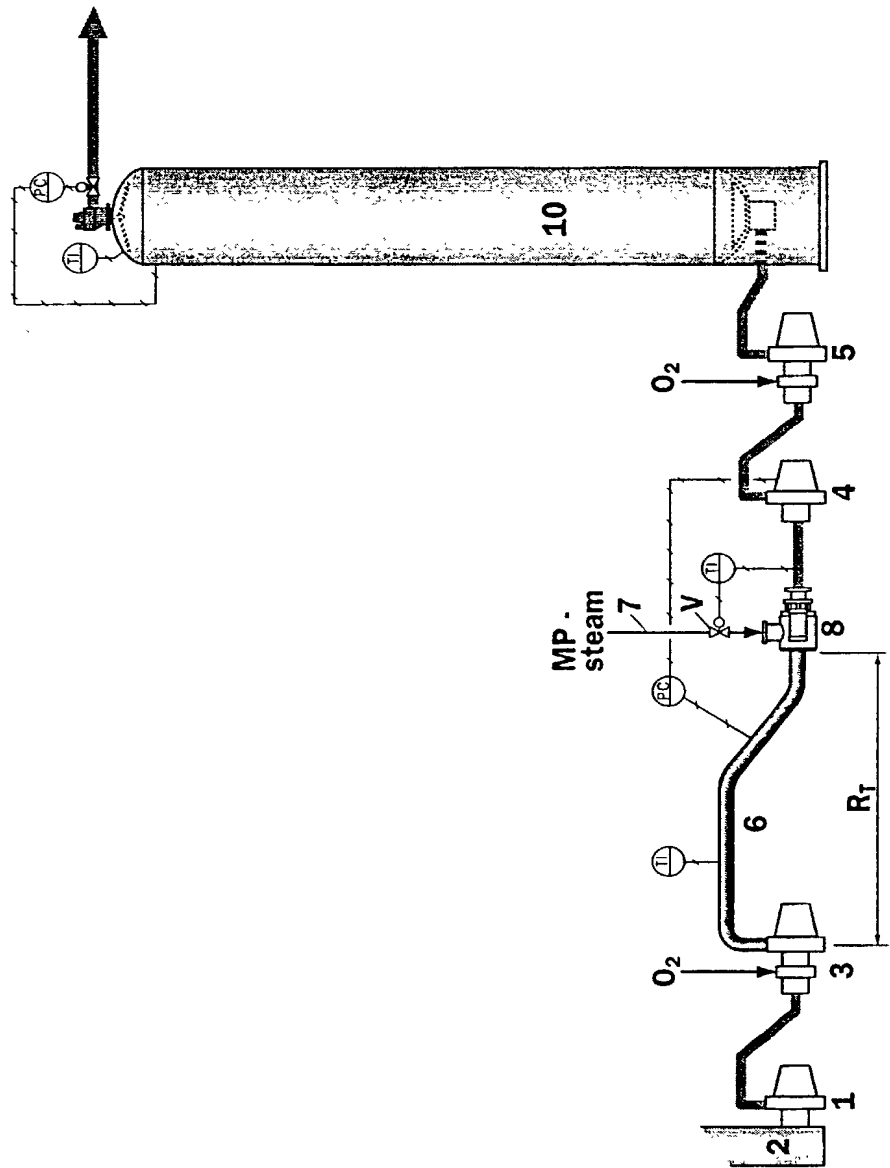
(54) Title: SYSTEM AND PROCESS FOR THE OXYGEN DELIGNIFICATION OF PULP CONSISTING OF LIGNOCELLULOSE-CONTAINING MATERIAL



(57) Abstract: The invention relates to a system and a process for the oxygen delignification of pulp which consists of ligno-cellulose-containing material and whose mean concentration is 8-18 % pulp consistency, in at least two stages. The invention is characterized in that oxygen delignification takes place in a process sequence consisting of: a first lamp (1), followed by a first oxygen mixer (3), followed by a first delignification zone (6) and, directly afthat, followed by a second steam mixer (8), followed by a second pump (4), followed by a third oxygen mixer (5) and, finally, a second delignification zone (10). This makes it possible, in an industrial process, to exploit the kinetics of oxygen delignification optimally in order to obtain selective oxygen delignification at low installation cost and low operating cost, even if only low-pressure steam is available.

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Fig1



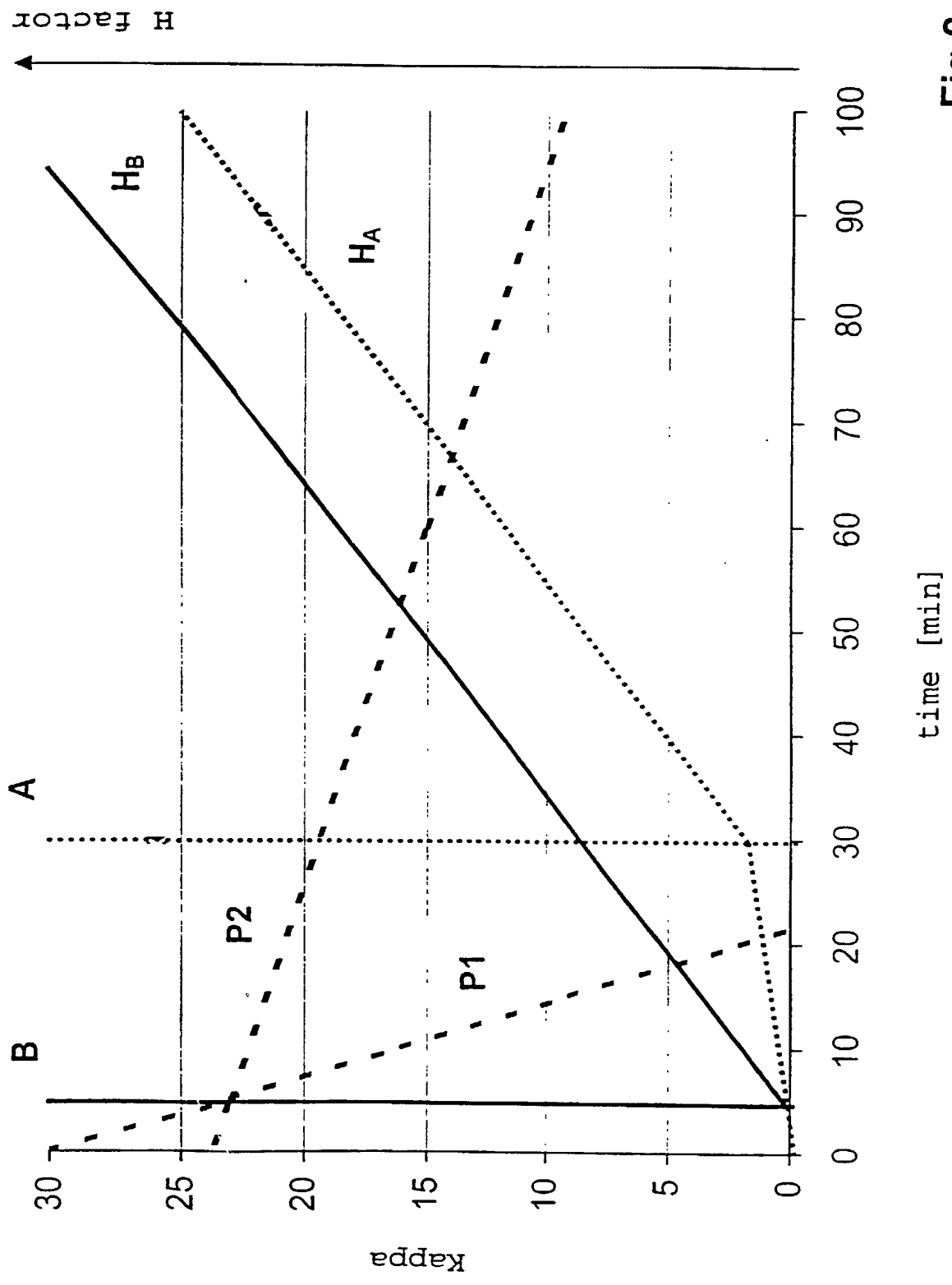


Fig.2

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§ 1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

<u>(not applicable)</u>	<u>(n/a)</u>	<u>(not applicable)</u>
(Application Serial No.)	(Filing Date)	(Status: patented, pending, abandoned)

The undersigned hereby authorizes Rolf Fasth, the U.S. attorney named herein, to accept and follow instructions from Kvaerner Pulping AB as to any action to be taken in the Patent and Trademark Office regarding this application without direct communication between Rolf Fasth and the undersigned. In the event of a change in the persons from whom instructions may be taken, Rolf Fasth will be so notified by the undersigned.

I hereby appoint Rolf Fasth, Registration No. 36,999, to prosecute this application, to file a corresponding international application, and to transact all business in the Patent and Trademark Office connected therewith.

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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**COMBINED DECLARATION AND POWER OF ATTORNEY
FOR PATENT APPLICATION**

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am an original, first and joint inventor of the subject matter which is claimed and for which a patent is sought on the invention entitled SYSTEM AND METHOD FOR OXYGEN DELIGNIFICATION OF PULP MADE FOR LIGNOCELLULOSIC MATERIAL, the specification of which was filed as International Patent Application No. PCT/SE00/01435 on 5 July 2000.

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations, § 1.56(a). If this is a continuation-in-part application filed under the conditions specified in 35 U.S.C. § 120 which discloses and claims subject matter in addition to that disclosed in the prior copending application, I further acknowledge the duty to disclose material information as defined in 37 CFR §1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of the continuation-in-part application.

I hereby claim foreign priority benefits under Title 35, United States Code, § 119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s)			Priority Claimed	
<u>9902586-8</u>	<u>Sweden</u>	<u>06 July 1999</u>	[X]	[]
(Number)	(Country)	(Day/Month/Year)	Yes	No

I hereby claim the benefit under Title 35, United States Code, § 120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, § 112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations,

RF:sj 11/30/01 128.833USN

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Inventor's signature

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